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SPECIFICATION

1. Title of the Invention

PRODUCING METHOD OF RUBBER-CONTAINING WAX

2. Claims

A producing method of a rubber-containing wax, in which a rubber or a rubber-wax master batch containing 50% by weight or more of a rubber component is added to waxes, followed by mixing and agitating at a temperature in the range of 80 to 300°C, at the maximum outer peripheral speed of an agitating blade in the range of 10 to 100 m/sec and at the maximum substantial agitation consumption power during a dissolution operation in the range of 30 to 800 Kw/t·product to produce a rubber-containing wax, in which 3 to 30% by weight of a rubber component is contained.

3. Detailed Description of the Invention

The present invention relates to a novel producing method of a rubber-containing wax obtained by mixing and dissolving a rubber and waxes. In more detail, the invention relates to a producing method of a rubber-containing wax, in which a rubber or a rubber-wax master batch containing 50% by weight or more of a rubber component is added to waxes, followed by mixing and agitating at a predetermined temperature, at an outer

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peripheral speed of an agitating blade and at an agitating power to produce a rubber-containing wax.

A rubber-containing wax where a rubber component and waxes are homogeneously dissolved and mixed was found to be excellent in that the elastic property of waxes could be increased and the water-proofness and wear resistance were improved more than an existing case where only waxes were used to be suitable as a waterproof material of, for instance, papers and clothes. However, the rubber-containing wax can be produced with difficulty from the viewpoint of the incompatibility of the waxes and rubber and an excellent producing method has never been found.

In this connection, the inventors have conducted a preliminary study of a producing method of a rubber-containing wax. In the first place, into 1-l beaker, 600 g of 180°C microwax is added, followed by heating and rendering the microwax a liquid at substantially 180°C, further followed by adding 20 pieces of heptyl rubber chips obtained by cutting a sheet of heptyl rubber to a magnitude of substantially 1 g per piece, still further followed by mixing and agitating as usual by rotating an agitating blade having a blade diameter of 40 mm at substantially 400 rpm. However, there was found no appearance of dissolution. Then, butyl rubber and 180° microwax were mixed by use of a roller so as to be 80/20 (by weight ratio), followed by cutting into dices each having a

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weight of substantially 1 g, further followed by adding into a beaker according to a manner similarly to that of the 180° microwax dissolved at 180°C, still further followed by similarly agitating at the temperature for substantially 6 hr. However, the mixture could not be dissolved. When such operations were conducted, a color of the microwax deteriorated with a heating time. From the experiences, it was found that a dissolution time when the rubber-containing wax is prepared had to be set short also from the product management.

As obvious from the above preliminary experiments, when small chips of rubber are added in a molten wax and mixed and agitated as usual, the rubber is not at all dissolved. Furthermore, even when small chips of a rubber-wax master batch containing a rubber component much are similarly added and agitated, the rubber was very difficult to dissolve.

Furthermore, when a rubber-wax master batch that contains a wax component much is used, only a slight amount thereof can be dissolved under ordinary mixing and agitation conditions. However, when a rubber-wax master batch containing for instance 50% by weight or more of a rubber component is used, particle diameters of the master batch have to be made smaller to make a surface area/volume ratio larger. Accordingly, a dissolution time becomes longer and the deterioration such as mentioned above is caused; as the result,

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in order to obtain a rubber-wax master batch less containing a rubber component (abundant in the was component), for instance, containing less than 50% by weight of the rubber component, the production cost becomes higher. Furthermore, when a wax component is increased, a master batch production by use of a Banbury mixer becomes impossible from the viewpoint of taking out the master batch from the mixer.

Then, the inventors have conducted many experiments and obtained a conclusion below. That is, it was found that, in order to dissolve a solid rubber or a rubber-wax master batch in waxes in a short time in an agitation dissolution bath, there was an obvious limit in an agitation bath mainly designed for so-called ordinary mixing. It was also found for the first time from various dissolution experiment results, that not by simply mixing and agitating but by applying shear pulverization to a rubber component that is put in a dissolution bath where waxes are present to simultaneously dissolve the pulverized rubber component in the waxes, the rubber can be dissolved in the waxes in a short time.

It was found that according to the method, a single rubber or a rubber-wax master batch in which the wax component is rather less contained can be dissolved in waxes in a short time and a rubber-containing wax that contains the rubber at a high concentration can be produced in a short time. From many experimental data, it was further found that, in order to

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effectively carry out a dissolution method such as the "shear pulverization dissolution", a peripheral speed of an outer periphery of an agitating blade of a dissolution bath and a necessary power substantially used in the agitation have to be predetermined values or more, thereby the invention comes to completion.

The invention relates to a producing method of a rubber-containing wax, in which a rubber or a rubber-wax master batch containing 50% by weight or more of a rubber component is added to waxes, followed by mixing and agitating at a temperature in the range of 80 to 350°C, at the maximum outer peripheral speed of an agitating blade in the range of 10 to 100 m/sec and at a substantial agitation consumption power during a dissolution operation in the range of 30 to 800 Kw/t·product to produce a rubber-containing wax, in which 3 to 30% by weight of a rubber component is contained.

The "waxes" here indicates petroleum wax, synthetic wax and natural wax and have the crystallinity or microcrystallinity of which melting temperature is in the range of 100 to 200°F. The petroleum wax is for instance paraffin wax, motor oil wax or microwax and one obtained by subjecting a distillation residue in a reduced pressure distillation, which is obtained for instance in a purification process of crude oil, or a mixture of heavy lubricant oil fraction and residue to a dewaxing process. In the invention,

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oil-containing waxes (for instance, slack wax) that contain an oil fraction that is an intermediate product when refined wax is obtained from petroleum, for instance, in the range of 5 to 10% by weight or less can be effectively used as well. Furthermore, the synthetic wax is popularly termed also as polyolefin wax and has an average molecular weight in the range of 500 to 10,000. The synthetic wax can be obtained by means of a method where ethylene and propylene are directly polymerized, a method where high molecular weight polyolefins are pyrolyzed to low molecular weight products or a method that makes use of low molecular weight polyethylene by-produced when polyethylene is produced. The natural wax can be naturally obtained and indicates bee wax, carnauba wax and so on.

The rubber in the invention is a natural rubber or a synthetic rubber. Examples of the synthetic rubbers include rubber-like materials such as SBR, polybutadiene, alfin rubber, polyisoprene rubber, BP rubber, EPDM rubber, polyisobutylene and butyl rubber. In the rubber here, a deterioration inhibitor usually contained in a rubber and other filler may be contained and various kinds of oils that dissolve or swell the rubber may be contained.

Furthermore, the rubber-wax master batch mentioned here, which contains 50% by weight or more of rubber component, is a master batch of a rubber and waxes, which contains 50% by

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weight or more of the rubber component. This is a master batch that can be readily obtained by mixing a rubber and waxes normally by use of a Banbury mixer or a roller mixer.

In the invention, the components are mixed and agitated at a temperature in the range of 80 to 300°C and preferably in the range of 120 to 220°C under predetermined conditions. When the temperature is 80° C or less than that, a sufficiently homogeneous product cannot be obtained. On the other hand, when the temperature is 300°C or more, raw material components tend to exhibit deteriorating tendency and high temperature devices are disadvantageously necessary.

In the invention, a mixing and agitation operation is carried out at the maximum outer peripheral speed of an agitation blade in the range of 10 to 100 m/sec and at the maximum substantial agitation consumption power during a dissolution operation in the range of 30 to 800 Kw/t·product. Here, the maximum substantial consumption power is a value obtained by subtracting from the maximum agitation consumption power during an agitation/mixing operation a consumption power when the agitation is carried out under the same agitation conditions with no load. When the maximum outer peripheral speed is smaller than 10 m/sec, and when the agitation consumption power is less than 30 Kw/t·product, a sufficiently homogeneous rubber-containing wax cannot be produced in a short time. Furthermore, when the maximum outer peripheral

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speed is larger than 100 m/sec and when the substantial agitation consumption power is larger than 800 Kw/t·product, an apparatus can be equipped with difficulty and at the same time it is difficult to inhibit heat from generating to be economically disadvantageous.

The foregoing agitation operation of the invention can be carried out in batch or continuously with a closed or partially opened agitation mixer with normally two or more agitation blades.

In a producing method of a rubber-containing wax of the invention, the raw material components are conveyed into an agitation mixer and agitation mixed under the conditions to produce the rubber-containing wax.

A composition of the rubber-containing wax obtained according to the invention contains the rubber component in the range of 3 to 30% by weight and preferably in the range of 5 to 25% by weight.

The rubber-containing wax preferably obtained according to the invention and containing a rubber component in the range of 5 to 30% by weight is normally liquid under an agitation temperature condition and can readily form, in this state per se, or by simply mixing with waxes in a heated and heat-melted state, or by line mixing, a rubber-containing wax that contains a rubber component in the range of 1 to 5% by weight and can be effectively used in the various applications.

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In what follows, the inventive method will be more specifically described with reference to examples.

Examples

A predetermined amount of rubber and a wax are agitation mixed under predetermined conditions as shown in a table and thereby a rubber-containing wax is prepared. According to a procedure of the agitation mixing, in the beginning, a predetermined amount of wax shown in (7) is put in a dissolution bath having a dimension shown in (2) and heated to 120°C. Subsequently, a predetermined amount of rubber is charged according to a charging method of rubber, which is shown in (5), so as to be a concentration shown in (6). The respective feeding amounts are calculated in advance from a total feeding amount shown in (9). After a rubber component is charged, an agitator is rotated up to an agitation blade peripheral speed shown in (10). Then, while maintaining a dissolution temperature as shown in (8), the agitation is continued for a dissolution time shown in (13), followed by stopping the agitation.

In the table, (1) denotes an experiment number; (2), a dimension and volume of a dissolution bath; (3), a diameter of an agitation blade; (4), kind of a rubber used; (5), a method of introducing a rubber component; (6), a rubber component concentration in a product; (7), used waxes; (8), a dissolution mixing temperature; (9), a total feeding amount; (10), the

maximum peripheral speed of an outer periphery of an agitation blade; (11), the maximum substantial agitation consumption power; (12), the maximum substantial agitation consumption power per 1 ton of product; (13), an agitating and mixing time; and (14), a melting state of a product rubber wax. The melting state is expressed with an amount of a rubber component remaining when the product rubber wax melt is passed through a 20-mesh filter. As obvious from descriptions of (14) in table, according to the method of the invention, a rubber wax can be very effectively produced.

Furthermore, to rubber waxes obtained in experiment Nos. 1 and 5, at substantially 150°C, a predetermined amount of 180° microwax is added, followed by manually mixing by use of an agitation rod, and thereby a rubber wax that contains a rubber component concentration at substantially 3% by weight can be readily obtained.

Notes in the table are as follows.

(Note 1) A rubber/wax master batch containing a rubber and 180° microwax at a ratio of 80: 20 is put in as a chip of substantially 5 g per piece.

(Note 2) Though same as the (note 1), a master batch is put in as a chip of substantially 50 g per piece.

(Note 3) A crude rubber is used per se. The crude rubber is put in as a chip of substantially 5 g per piece.

(Note 4) A crude rubber is used per se. The crude rubber is

put in as a chip of substantially 50 g per piece.

Table (1)

(1) Example No.

Comparative Example 1, 1, 2, 3, Comparative Example 2, 4, 5, 6

(2) Dissolution Bath Specification

Inner Diameter (mmϕ) 230, 230, 230, 230, 230, 230, 230, 230

Depth (mm) 220, 220, 220, 220, 220, 220, 220, 220

Volume (l) 9, 9, 9, 9, 9, 9, 9, 9

(3) Diameter of Agitation Blade (mm)

220, 220, 220, 220, 220, 220, 220, 220

(4) Kind of Rubber

Butyl Rubber BESO-218, Butyl Rubber BESO-218, Butyl Rubber BESO-218, Butyl Rubber BESO-218, Butyl Rubber BESO-035, Butyl Rubber BESO-035, EpT Rubber Mitusi-EpT-1045, JSR-1500

(5) Adding Method of Rubber

(Note 1), (Note 2), (Note 2), (Note 2), (Note 3), (Note 4), (Note 4), (Note 4)

(6) Rubber Concentration (% by weight)

20, 20, 20, 20, 20, 20, 20, 20

Table (2)

(1) Example No.

Comparative Example 1, 1, 2, 3, Comparative Example 2, 4, 5, 6

(7) Used Waxes

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Nippon Oil Corporation 135° Paraffin, Nippon Oil Corporation 180° Microwax, Nippon Oil Corporation 145° Paraffin, Nippon Oil Corporation 135° Paraffin, Nippon Oil Corporation 180° Microwax, Nippon Oil Corporation 180° Microwax, Nippon Oil Corporation 180° Microwax, Nippon Oil Corporation Microslack Wax

(8) Temperature (°C)

185, 200, 185, 185, 180, 180, 180, 180

(9) Total Feed Amount (kg)

4, 4, 4, 4, 5, 5, 5, 4

(10) Peripheral Speed of Agitation Blade (m/sec)

3.5, 25.2, 13.8, 37.8, 3.5, 26.4, 26.4, 25.2

(11) Maximum Substantial Agitation Consumption Power (kW)

0.1, 1.6, 0.4, 2.2, 0.1, 1.5, 1.6, 1.5

(12) Same as left (KW/t Product)

25, 400, 100, 560, 20, 300, 320, 300

(13) Dissolution Time (min)

480, 40, 180, 30, 720, 60, 60, 300

(14) Dissolution State

Fault, Completely Dissolved, Completely Dissolved, Completely Dissolved, Not Dissolved, Completely Dissolved, Completely Dissolved, Completely Dissolved